A JEWEL MOUNTED MICRO-BALANCE FOR THE MEASUREMENT OF MAGNETIC SUSCEPTIBILITIES OF CRYSTALS

(Miss) D. DAS

DEPARTMENT OF MAGNETISM,
INDIAN ASSOCIATION FOR THE CULTIVATION OF
SCIENCE, CALCUTTA-32.

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ABSTRACT. A jewel-mounted microbalance has been designed and constructed for the measurement of the magnetic susceptibilities of the dia- and paramagnetic crystals at various temperatures The detailed description and method of calibration of the balance are given in the paper. The balance is of robust and stable construction and its deflectional sensitivity is magnified very much by a suitable optical lever arrangement The magnetic force upon a sample suspended by a thin fibre from one arm of the balance is compensated by the force exerted upon a small current bearing coil by a permanent magnet The sample when in the form of a single crystal is free to set with its maximum susceptibility in the horizontal plane along the direction of the magnetic field When the balance is calibrated by a standard sample the above method gives directly the maximum susceptibility in the horizontal plane. The sensitivity of the compensation device is 1.65×10^{-6} gms per microampere of compensating current

INTRODUCTION

Of the methods (Krishnan and Banerji, 1933, Dutta, 1944; Dutta Roy, 1955; Bates, 1961) which are being utilized here as well as elsewhere for the determination of the magnetic susceptibilities of different types of para- as well as diamagnetic substances particularly of the single crystals of these, the quartz fibre micro-balance method devised by Bose (1947) has been found to be very convenient, though somewhat less sensitive than some of the other methods, namely, those working on the principle of a Curie balance. In Bose's method the single crystal suspended freely, with a very fine fibre from one arm of the balance, in a horizontal magnetic field with a vertical gradient sets with its maximum susceptibility in the horizontal plane along the field and at the same time moves along the gradient, thus eliminating the uncertainty about the direction of measurement of the susceptibility and affording obvious economy of space in the design of cryostats and heaters.

But the quartz suspension system was very sensitive to disturbances and liable to frequent breakage. Attempts to replace quartz fibre with phosphor-bronze strip did not improve matters and replacement became difficult owing to unavailibility of the strips. Moreover, in horizontal fibre suspension systems there was always sagging of the strips and the usual yielding at the points of attachment. Finally, balancing of magnetic force by manual torsion of the fibre introduced unwanted disturbances in the system and the sharp magnetic gradient used led to uncertainties of calibration With the object of removing all these objections the manufacture of a jewel mounted micro-balance and other concomitant arrangements were undertaken which will be described in details in the present paper.

THEORY

When a small magnetic crystal is placed in an inhomogeneous magnetic field, it experiences a couple due to its magnetic anisotropy, given by (Nye, 1957)

$$G_{ij} = v(-k_{ik}H_kH_j + k_{jk}H_kH_i) \qquad (i, j, k = 1, 2, 3) \qquad . \qquad (1)$$

where k's and H's are the components of the volume susceptibilities and mangetic field strengths respectively and v the volume of the crystal.

In addition to this the crystal experiences a translational force given by (Nye 1957)

$$F_i = \tfrac{1}{2}v(k_{jk}-k_0) \cdot \frac{\partial}{\partial x_i}(H_j H_k) \qquad (2)$$

where k_0 is the volume susceptibility of the surrounding medium

If now the experimental arrangement is made such that the magnetic field is in the x_1 direction (horizontal direction from pole to pole) and the gradient in the x_3 direction (vertical), the couple due to magnetic anisotropy will be in the $x_1\,x_2$—plane (horizontal) and about x_3 and the translational magnetic force will be along the x_3 direction. If the experimental set up be so arranged that the crystal is incapable of taking up rotation about any direction except x_3 the major couples due *to anisotropy of shape* about x_1 and x_2 become totally ineffective. A small residual couple of this type about x_3 may be eliminated by grinding the crystal in the form of a disc in $x_1\,x_2$ plane.

Then the *magnetic anisotropy* which is only effective for the purpose of rotating the crystal in the plane $x_1\,x_2$ will be given by

$$\tfrac{1}{2}v(k_{max}-k_{min})\,H^2 \sin 2\psi \qquad \ldots \quad (3)$$

where k_{max} and k_{min} are the maximum and minimum values of the volume susceptibilities in the $x_1\,x_2$ plane and ψ is the angle between the magnetic field and k_{max}. If the crystal is perfectly free to rotate about x_3, the above couple due to magnetic anisotropy will place k_{max} direction practically along the field (x_1 direction) and at the same time crystal will experience a force due to the gradient in the x_3 direction given by

$$F_3 = v(k_{max}-k_0)\,H_1\frac{\partial H_1}{\partial x_3} \qquad (4)$$

The measurement of this force will then give us the maximum value of the magnethe susceptibility in $x_1\,x_2$ plane. The above principle is utilised in the balance described below.

Description of the balance

(a) The balance consists of a light horizontal beam made from thin aluminium sheet about 5 millimeters wide and 25 centimeters in length bent along the longitudinal axis to an L-shaped cross section serrated along the vertical section, to take movable wire riders.

The beam is capable of rotation in a vertical plane about a thin non-magnetic stainless steel spindle rigidly fixed at right angles (horizontally) through the balance beam For sensitiveness in detecting the movement of the beam the spindle is fixed at a point about 18 cm from the end of arm at which the sample is suspended. The spindle is accurately mounted on a pair of jewel bearings set in a U-shaped brass holder and fixed with arms upright on a horizontal aluminium base provided with levelling screws The jewel bearings consist of a pair of perforated jewels with a pair of flat jewels behind them to prevent any lateral motion of the spindle. The jewels are set within two accurately cut screw heads passing co-axially through threaded holes near the top of the two arms of the brass holder and locked in position by locking nuts.

(b) *Method of observation of the movement of the beam*

The movement of the balance beam could be observed as usual by mounting a vertical mirror upon the spindle, but for very small forces the movements of the beam are not perceptible with this arrangement. Consequently, a simple device for magnifying these movements has been adopted A small light plane mirror has been kept vertical by means of two horizontal stretched unspun silk fibres attached at the two ends of the horizontal diameter of the mirror. The position of the mirror is about 3 cm above the horizontal balance beam near its end from which samples are suspended for magnetic measurements. A small piece of glass rod about 1 cm in length is attached with the mirror at right angles to its plane so that in undisturbed position the rod is always horizontal and just above the balance beam. A small piece of quartz fibre is attached between the balance beam and the small horizontal rod with the mirror in such a way that the fibre is always taut. Thus any very feeble movements of the beam is now many times magnified and can be observed by lamp and scale arrangement.

(c) *Suspension system*

The suspension system consists of three parts attached successively to one another (i) the upper portion consisting of a thin and short glass rod hooked at the upper end to hang from the balance arm, (ii) the central portion consisting of a fine quartz fibre of sufficient length, and (iii) the lowermost portion consisting of a thin long pyrex rod to the lower end of which is fixed the crystal, with the known direction vertical and lying accurately in the small region over which the magnetic force is appreciably constant The pyrex rod is of such a length that the thin index rod fixed at its upper end shows above the heater or the cryostat chamber, between the pole pieces, within which the crystal is suspended for taking measurements at high or low temperatures.

(d) *Method of measurement of the magnetic force on the sample*

It is obvious that the specimen suspended with the fine quartz fibre from one arm of the micro-balance in the non-homogeneous magnetic field, between Sucksmith (1939) type of pole shoes over the flat pole pieces of the electromagnet (as adopted here by Dutta Roy 1955), sets with its maximum susceptibility in the horizontal plane, along the horizontal magnetic field and at the same time moves bodily along the vertical gradient The magnetic translational force is measured by an electrodynamic arrangement placed at the other end of the balance beam. The arrangement consists of a small coil of 70 turns of enamelled copper wire of 42 s w.g wound on a light hollow cylindrical perspex former (about 1 cm in length and 8 mm in diameter) suspended from the end of the balance beam and placed inside the magnetic field of a permanent magnet. Pole pieces of the permanent magnet are shaped to concave cylinders and the coil hangs freely concentrically between them When a current is passed through the coil, the balance beam experiences the usual electrodynamic force proportional to the current The terminals from the coil are taken in such a way that very little restriction is imposed on the movement of the balance beam

The entire balance system consisting of the above described parts is placed firmly screwed over a plane base provided with levelling screws. The entire system is covered by a perspex cover to protect it from draught and dust. There is an arrangement fixed to the base by which the beam can be kept arrested from outside the case when not in use. The specimen to be studied is suspended from one end of the balance beam, passes through a hole in the base into the inhomogeneous magnetic field and is counterpoised by placing riders on the beam.

When the magnetic field is switched on, the beam is deflected due to the magnetic force and can be restored to the original position by sending a suitable current through the balancing coil. The description of the different parts can be better followed by referring to the adjoining diagram (Fig. 1.)

Test of sensitiveness :

In order to test the sensitiveness etc. of the balance, the beam was first made free and horizontal and an accurately weighed rider made of thin aluminium wire is placed successively at different points on the balance beam starting from one extreme end. The corresponding deflections of the mirror was observed by a lamp and scale arrangement and were balanced by sending suitable currents through the balancing coil. The current was observed in an accurate, sensitive and calibrated microameter of which readings are correct to ±0.5 microampere. The balancing current was more accurately measured by recording the drop of potential

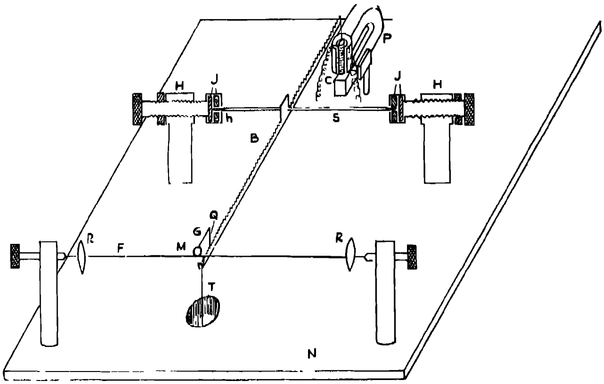

Fig. I Schematic diagram of the balance (not to scale).
B ————Arm of the balance. S———— Stainless Steel Spindle.
J, J————Jewels, H, H————Brass holders. P————Permanent Magnet.
C————Current bearing coil. M————Light plane mirror.
G————Pyrex glass rod. Q————Quartz fibre. F————Unspun Silk fibre.
R, R————Copper springs. T————Suspension system.
N————Aluminium base of the balance.

across a standard resistance placed in the circuit, with the help of a *L.* and *N.* student's type potentiometer while the balancing current was flowing through the circuit. The results of observation are shown in Table I and the variation of current with force shown diagrammatically in Fig. 2.

TABLE I

Weight of the rider in gm	Position of the rider from the spindle in cm.	Effective load at the end of the beam in milligram.	Balancing current in micro-amperes	Sensitiveness in gm/micro-ampere
.00165	0	0	0	1.65×10^{-6}
	3 6	333	205 0	
	7 2	.667	405 0	
	10 8	1.00	610 0	
	14.4	1.32	810.0	
	18 0	1.65	1000.0	

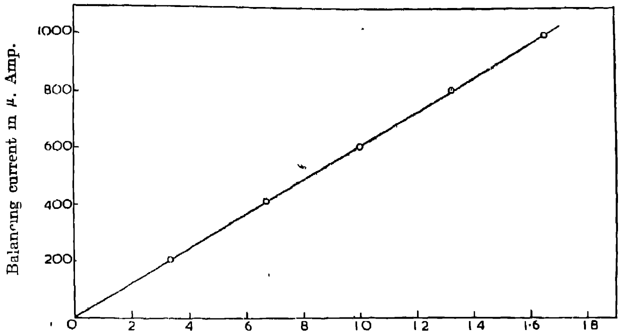

Fig. 2, Sensitivity and accuracy of the balance as shown by the variation of the balancing current with effective load at the end of the beam

It is observed that the relation between force and balancing current is a linear one It is further observed that with the present arrangement the sensitiveness obtained is about 1.65×10^{-6} gm/micro-ampere

Calibration and Standardisation .

Let a sample of mass m_1 and volume v_1 attached at the end of the suspension system of the balance experience a magnetic force F_1 balanced by a current i_1 and F_2 and i_2 be the corresponding force and current when the above specimen is placed by a standard specimen of mass m_2 and volume v_2, then

$$\frac{F_1}{F_2} = \frac{i_1}{i_2} = \frac{v_1}{v_2}\,\frac{k_1 - k_0}{k_2 - k_0}$$

where k_1 and k_2 are the volume susceptibilities of the specimen and the standard substance respectively, k_0 that of the medium in which the samples are placed which is generally air, provided the volumes are comparable and such that $H\dfrac{\partial H}{\partial x}$ over them is constant The mass susceptibility of the specimen will then be given by $\chi_1 = \dfrac{i_1}{i_2}\,\dfrac{m_2}{m_1}\left(\chi_2 - \dfrac{k_{air}}{\rho_2}\right) + \dfrac{k_{air}}{\rho_1}$ where ρ_1 is the density of the specimen χ_2 and ρ_2 are the mass susceptibility and density respectively of standard sample $k_{air} = .028 \times \left(\dfrac{300}{T}\right)^2 \times 10^{-6}$ at T° absolute For the measurement of paramagnetic susceptibilities pure crystals of chromium potassium alum have been taken as the standard substance because its susceptibilities at different temperatures have been very accurately measured here and elsewhere (de-Haas and Gorter 1929, Mme. Serres 1932, Dutta Roy 1958).

Mean gm. molecular susceptibility of chromium potassium alum taking the average of these values is given by $\chi_M = 6080.5 \times 10^{-6}$ at 306.8°K.

In the case of diamagnetic samples, conductivity water is taken as the standard substance of which mass susceptibility $\chi = .7225 \times 10^{-6}$ at 30°C.

In order to check the reliability of the balance a number of measurements have been made for paramagnetics with pure crystals of ferric ammonium alum (cubic) and $NiSO_4 . 6H_2O$ (tetragonal) grown from aqueous solutions and for diamagnetics with pure crystals of sodium chloride and potassium chloride (both cubic) obtained from Harshaw Chemicals, (U S A) The detailed results of measurements are given in the adjoining tables (Tables II and III).

TABLE II

Paramagnetic Samples

Substance	Mass, density and balancing current corrected for pull on carrier	Mass susceptibility $\chi \times 10^6$ c g s.e m u	P_{eff} in Bohr Magnetons at 300°K of unknown substances	
			Present value	Earlier values
Chromium potassium alum (standard)	$m = .1088$ gm $\rho = 1.842$ gm/cc $i = 77.0$ μ A.	11.678 at 306.8°K		34.81 (Dutta Roy 1958) 35 (Spin only value)
Ferric ammonium alum (unknown)	$m = .1000$ gm $\rho = 1.724$ gm/cc $i = 175.2$ μ A	28.89 at 306.8°K	34.80	34.79 (Onnes and Oosterhuis 1926) 34.78 (Mitra 1963)
Chromium potassium alum (Standard)	$m = .0680$ gm $\rho = 1.842$ gm/cc $i = 51.0$ μ A	11.727 at 305.5°K		9.687 (Dutta Roy 1958) 9.656 (Mookherjee 1946)
$NiSO_4, 6H_2O$ (along tetragonal axis) (unknown)	$m = .1226$ gm $\rho = 2.080$ gm/cc $i = 114.5$ μ A	14.598 at 305.5°K	9.677	9.701 (Mitra 1963)

TABLE III

Diamagnetic Samples

Substance	Mass density and balanceing current corrected for pull on carrier at 30°C	Mass susceptibility $\chi \times 10^6$ c g.s.e.m.u	Earlier values of Mass susceptibility $\chi \times 10^6$. c g s e.m u
Water	$m = .1232$ gm. $\rho = .99567$ gm/cc at 30°C $i = 105.3$ μ.A	.7225 at 30°C	
Crystals of sodium chloride	$m = .1366$ gm $\rho = 2.165$ gm/cc $i = 85.0$ μ A.	.5186	.5183*
Crystals of patassium chloride	$m = .1198$ gm $\rho = 1.984$ gm/cc. $i = 75.0$ μ.A.	.5228	.5230*

*Tables de constantes et données numériques 1957, Edited by G. Foëx *et al*, Vol. 7, "Diamagnétisme et Paramagnétisme".

Concluding Remarks :

The lever arrangement for magnification of the deflections could be replaced by some other arrangement such as a balanced photo-cell system Mitra *et al*, 1963 but since this is not easily available here we are to remain content with the present system which is quite simple yet very efficient

It would be interesting to mention in this connection that the present arrangement can easily be utilised with slight modification for the measurement of magnetic anisotropy and maximum absolute susceptibilities in the horizontal plane of a single crystal with the same setting Only extra arrangement that is necessary is for giving a vertical motion of the magnet by jack-screw so as to bring the crystal in the homogeneous part of the Sucksmith type pole gap and to provide a rotation of the magnet about the vertical suspension axis so that the magnetic couple may balanced against the torsion of the fibre. From this the anisotropy is calculated For the susceptibility the crystal is placed in the centre of the inclined parts of the Sucksmith gap as already explained. The arrangement is in course of being set up in our laboratory and the details of it will be published in due course.

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